Anupop Sasook 2008: Characterization of Silica Monolithic Column by Inverse Gas Chromatography. Master of Science (Chemistry), Major Field: Chemistry, Department of Chemistry. Thesis Advisor: Assistant Professor Orapin Chienthavorn, Ph.D. 101 pages.

Silica monolithic material having single piece skeleton with large through pore size was synthesized by an in situ reaction inside capillary tubing. Surface area of the silica monolithic material was determined by using an inverse gas chromatographic technique (IGC). To be characterized, the column was used as a sample connected between the injection port and the flame ionization detector via a fused silica tubing. Parameters affecting surface area determination including operating temperature, injection volume for each column, type of probe molecules were optimized. Selected probe molecules in this work were octane, nonane, decane and undecane of which optimum operating temperatures were 100, 120, 140 and 160˚C, respectively. All probe molecules could be used to determine the surface area with reproducible results. However, as decane gave minimum relative standard deviation, it was used as a probe molecule in this work. Parameters of column fabrication affecting specific surface area of the monolith were also studied. Results showed that specific surface area decreased with increasing column diameter and length. In addition, surface areas of silica monolithic columns determined in this work varied from approximately 10 to 300 $m^2 \cdot g^{-1}$, indicating unreproducible columns. To be used for separation as a typical gas chromatographic column, the surface of silica monolithic column was coated with polyethyleneglycol (PEG) (MW 10,000 $g \cdot mol^{-1}$). Three coating methods, namely evaporation, dynamic and static coating method, were studied. Best column efficiency for separating a mixture of hexane and acetone was found for a 0.32 mm × 43 cm column coated with 30% w/w PEG in acetonitrile by a static coating method. The resolving power of the column was corresponded to ca. 40 m. commercial Carbowax column with a shorter analysis time.

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